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13. ABSTRACT *(Maximum 200 words)*

In the final phase of this contract a new method for direct determination of strains in amorphous thin films using Grazing Incidence X-ray Scattering (GIXS) data of is described. Results for the residual stress distributions for sputter deposited B4C and SiC are compared to results obtained from more traditional x-ray methods such as Double Crystal X-ray Topography (DCDT). This new approach directly measures quantities intrinsic to the film independent of the type of substrate used for the coating. This allows measurements on complex surfaces ordinarily encountered in engineering applications. The GIXS-based technique yielded somewhat larger strains than the DCDT technique. The implications of these results will be reported in a series of future articles now in preparation.

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ULTRAHIGH STRENGTH-TOUGH SURFACE COATINGS VIA CONTROLLED NANOSTRUCTURES

FINAL REPORT:
March 1, 1995– February 28, 1999

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John C. Bilello and Steven M. Yalisove

U.S. ARMY RESEARCH OFFICE
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ULTRAHIGH STRENGTH-TOUGH SURFACE COATINGS VIA CONTROLLED NANOSTRUCTURES

By

John C. Bilello and Steven M. Yalisove

Abstract

In the final phase of this contract a new method for direct determination of strains in *amorphous* thin films using Grazing Incidence X-ray Scattering (GIXS) data of is described. Results for the residual stress distributions for sputter deposited B_4C and SiC are compared to results obtained from more traditional x-ray methods such as Double Crystal X-ray Topography (DCDT). This new approach directly measures quantities intrinsic to the film independent of the type of substrate used for the coating. This allows measurements on complex surfaces ordinarily encountered in engineering applications. The GIXS-based technique yielded somewhat larger strains than the DCDT technique. The implications of these results will be reported in a series of future articles now in preparation.

Project Report

Overview:

The study of stress and strain has been an important issue in thin film research since the early work of Stoney.[1] Measurements of strain in thin films have usually been carried out by coating a thin substrate and measuring the change in its curvature. These strain values are then converted to stress using linear elasticity theory. These techniques are highly sensitive ($\Delta d/d_0 \approx 10^{-7}$ and can perform measurements on films as thin as a few monolayers, but they measure the physical, or lattice, curvature of the substrate, not an intrinsic property of the film. They also require that the deposition be performed on specific flat substrates, typically Silicon wafers, which may be very different in nature from the desired material required in a particular engineering application.

Direct measurements of strain in crystalline thin films has been done using peak shifts via X-ray scattering experiments.[2,3] It has been shown that under certain circumstances there is a difference between these direct measurements and indirect ones made using substrate curvature techniques. Such studies, naturally, have been restricted to crystalline thin films. In this final report we will summarize a new method we developed for direct determination of strains in the amorphous thin films of B_4C and SiC. Substrate curvature measurements made using DCDT are presented for comparison. Earlier

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[1] G. G. Stoney, Proc. Roy. Soc. (London) **A82**, 172 (1909).
[2] S. G. Malhotra, Z. U. Rek, S. M. Yalisove, and J. C. Bilello, Thin Solid Films **301** (1-2), 45 (1996).
[3] I. C. Noyan and J. B. Cohen, *Residual Stress: Measurement by Diffraction and Interpretation* (New York: Springer-Verlag, 1987).

work in this project has been presented in other reports and will not be repeated herein.

Measuring strains in amorphous thin films depends on the principle that an X-ray diffraction scan is the Fourier transform of the average structure of the sample. Structural studies on bulk amorphous materials by reverse Fourier transform are well established, but have not been extensively used in thin films because of their inherently low scattering power[4,5]. This work addresses this issue through the use of synchrotron radiation.

Experimental Procedure:

Boron carbide and silicon carbide films of thickness 2520 ±50Å (B_4C) and 800 ± 20Å (SiC) were fabricated by DC and RF magnetron sputtering. The substrates were 100 mm wafers of (100) oriented Si cut into quarters.

Symmetric and asymmetric mode Grazing Incidence X-ray Scattering (GIXS) experiments were carried out on Beamline 7-2 at Stanford Synchrotron Radiation Laboratory (SSRL), at nominally 3 GeV and 100 mA at fill using an incident energy of 10 keV (λ = 1.2395Å). This wavelength was calibrated using a LaB_6 standard during each experimental run.

Incident angles in both GIXS modes were chosen in relation to the critical angles for total external reflection of 10keV x-rays. Indices of refraction for bulk materials were calculated based on stoichiometric

[4] B. E. Warren, *X-ray Diffraction* (Addison-Wesley, 1969).
[5] A. C. Wright, J. Non-Cryst. Solids **179**, 84 (1994).

composition and crystal lattice volume (for SiC and Si) or published densities (for B_4C). Refractive indices for the thin films were calculated based on the bulk values adjusted for estimated density. Incident angles in both GIXS modes were chosen just larger than the critical angles.

To determine the average interatomic spacings in the film, the intensity data per unit scattering volume was analyzed using the approximate method for polyatomic materials.[4] Tabulated atomic scattering factors and inelastic intensities were used and, to first order, stoichiometric compositions were assumed.[6] The average atomic scattering factor "f" was taken as the formula sum of individual scattering factors divided by the number of atoms, i.e. $K_m = Z_m$. The data was corrected and normalized to yield a reduced intensity i(**k**); a convergence factor $\alpha = 1$ was applied and a Fourier transform was performed. The result gave the reduced correlation function, in which the nearest neighbor peak locations were measured,[7] and also gave the radial distribution function (RDF). These real space distributions were averaged over the formula unit; they did not represent relationships between particular chemical species, for example, Si-Si correlations in SiC. To separate those 'partial' correlation functions and RDFs for each compound would have required three independent data sets in which the scattering power of the different elements varied, for example using X-ray anomalous scattering[8] or neutron scattering.[9]

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[6] International Tables for Crystallography, 2nd. ed., Kluwer Academic Publishers, 1987.
[7] Ch. Hausleitner and J. Hafner, Phys. Rev. B **47** (10), 5689 (1993).
[8] P. H. Fuoss, P. Eisenberger, W. K. Warburton, and A. Bienenstock, Phys. Rev. Lett. **46** (23), 1537 (1981).
[9] A. J. Leadbetter, J. Non-Cryst. Solids **179**, 116 (1994).

In-plane stress in the samples was measured using DCDT. The curvatures of the crystal lattices of the Si substrates were determined and the stress σ_{xx} found from the modified Stoney equation,

$$\sigma_{xx} = \frac{E_s(\kappa - \kappa_0)t_s^2}{6(1-\nu_s)t_f}, \qquad (1)$$

where κ-κ_0 is the curvature change, E_s, t_s, and ν_s are the modulus, thickness, and Poisson's ratio of the Si substrate, respectively, and t_f is the film thickness.

Experimental Results and Discussion:

Typical results for synchrotron symmetric and asymmetric scans are shown in Fig. 1 below. The extremely high intensity of the wiggler beam line 7-2 at Stanford Synchrotron Radiation Laboratory allowed excellent data collection rates and low signal-to-noise. Figure 1a shows three asymmetric (solid line) and three symmetric (dashed) GIXS scans while figure 1b shows one of each

The reduced correlation functions derived from symmetric and asymmetric GIXS experiments on B_4C are shown in figure 2. The plots in figure 2 (a) - (c) correspond to the asymmetric GIXS data sets in figure 1a, while (d) - (f) correspond to the symmetric mode data sets of figure 1a.

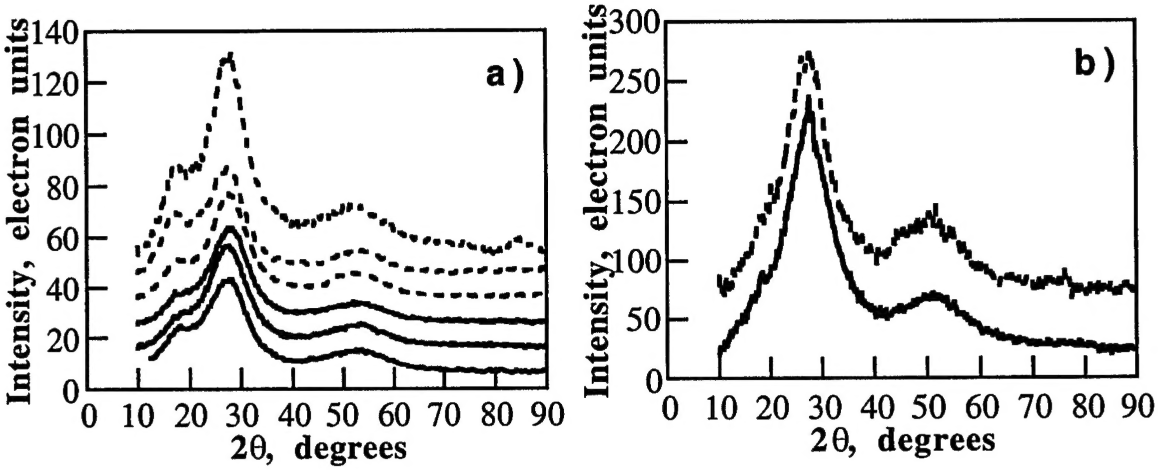

Figure 1. Asymmetric and symmetric GIXS scans of 2520Å B_4C (a) and 800Å SiC (b) thin films on Si substrates are shown. The symmetric scans are shown as dotted lines. Figure (a) contains six separate data sets offset by 10 e.u. while figure (b) contains two scans offset by 50 e.u. for clarity. Contributions from air/He and substrate scattering have been subtracted and the response has been scaled to the curve of average atomic scattering factor squared plus inelastic scattering. The wavelength λ = 1.2395Å (10 keV) was used.

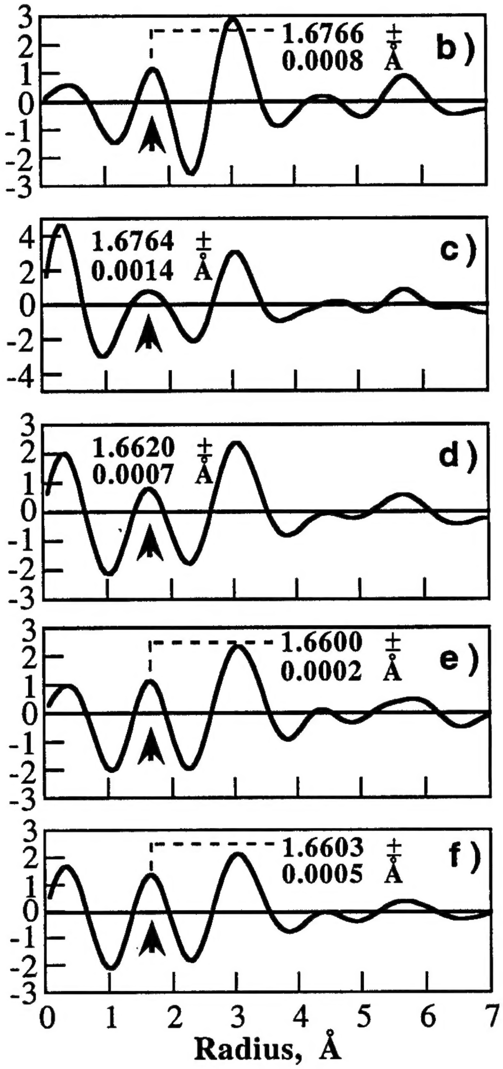

Figure 2. The reduced correlation functions $4\pi r(\rho(r)-\rho_o)$ (atoms per $Å^2$) resulting from GIXS of B_4C on Si are shown. Figures (a) - (f) correspond to the six data sets in figure 1 (a) and to the first six experiments listed in Table I. Asymmetric mode results are in (a) - (c) while symmetric are in (d) - (f). In each figure, the average first nearest neighbor distance or interatomic spacing is indicated.

The x-axis in the <u>reduced</u> correlation function plots represents radial distance from the average scattering atom. The y-axis depicts the density function $\rho(r)$, which represents the average number of atoms per

unit volume at distance r from the average scatterer. Analogous results were found for SiC films.

Radial distribution function (RDF) plots are presented for SiC in figure 3 and similar results were found for B_4C. In figure 3 (a) are the results from asymmetric scans are shown, and in figure 3 (b) are symmetric results.

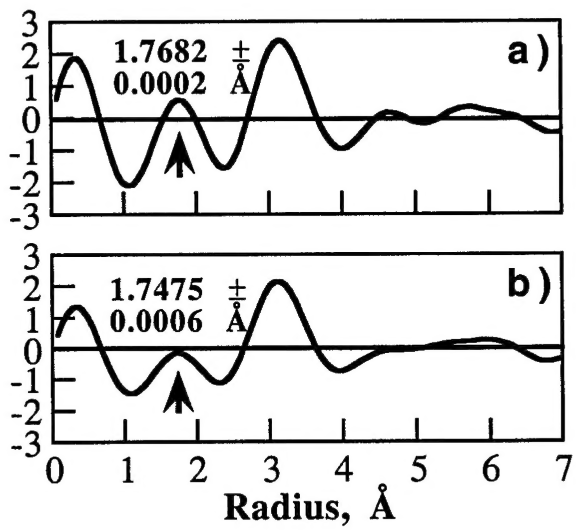

Figure 3. The reduced correlation functions $4\pi r(\rho(r)-\rho_o)$ (atoms per $Å^2$) resulting from asymmetric (a) and symmetric (b) GIXS of SiC on Si are shown. The plots correspond to the data sets in figure 1 (b. The average first nearest neighbor distance or interatomic spacing is indicated by the arrow.

The x-axis of each plot, "r", represents distance from the average scattering atom, and the formula for the y axes is given in the figure captions. The average density of the material is shown as a dotted line. The radial distribution function can be integrated through a range of r-values to give an average coordination number and is the most common representation of the data. By comparing the measured "r" values with those determined in the

relaxed case one can determine the strains. By repeating this exercise for a variety of incident x-ray penetration angles one can assess the complete strain tensor in a manner analogous the Ψ^2 squared method used for crystalline materials. These relative strain data are then converted to residual stress using linear elastic analysis. The results are shown in Fig. 4. A detailed discussion of these experiments, including a complete error analysis is in progress and will be the subject of a future publication.

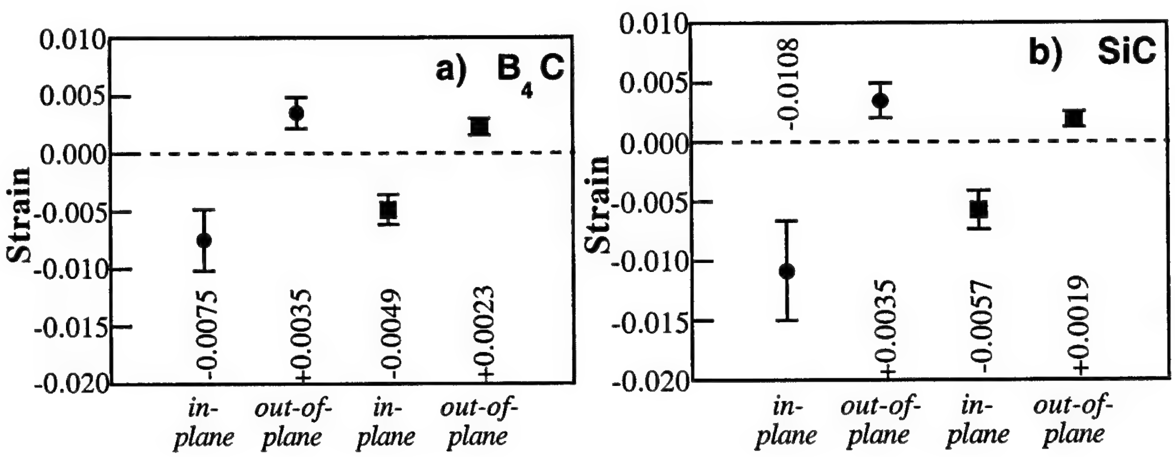

Figure 4. Strain results for B_4C (a) and SiC (b) thin films on Si are shown for the GIXS and DCDT techniques in two directions relative to the substrate: *in-plane* and *out-of-plane*. Results from GIXS are shown as circles on the left of each plot and DCDT results are shown as squares on the right of each plot. Numerical strain values are included.

Summary

Strains in thin films of amorphous B_4C and SiC have been measured using synchrotron Grazing Incidence X-ray Scattering in the symmetric and asymmetric modes. The results were compared to observations from substrate curvature measurements using Double Crystal Diffraction

Topography. For the first time, use of the GIXS technique permitted assessment of strains in amorphous thin films without reliance on indirect substrate curvature methods. This method can readily measure strains on practical substrates for which curvature measurements are in practice virtually impossible.

APPENDIX I

PUBLICATIONS DURING PERIOD:
March 1, 1995 to February 28, 1999

Articles in Refereed Publications:

1. *Residual Stress, atomic structure, and growth morphology in B4C/SiC multilayer coatings*, J. Hershberger, T. Ying, F. Kustas, L. Fehrenbacher, S. M. Yalisove and J. C. Bilello, Surface and Coating Technology, **86-87**, (1996), 237-242.

2. *Depth dependence of residual strains in textured Mo thin films using high resolution x-ray diffraction*, S. G. Malhotra, Z. U. Rek, S. M. Yalisove, and J. C. Bilello, J. Appl. Phys., **79**(9), (1996), 6872-6879.

3. *Growth and Characterization of Ta/W Multiscalar Multilayer Composite Films*, A. K. Malhotra, S. M. Yalisove and J. C. Bilello, Thin Solid Films, **286**, (1996), 196-202.

4. *Growth Anisotropy And Self-Shadowing: A Model For The Development Of In-Plane Texture During Polycrystalline Thin-Film Growth*, O. P. Karpenko, J. C. Bilello, S. M. Yalisove, J. Appl. Phys. **82**:3 (1997) 1397-1403.

5. *Analysis Of Thin Film Stress Measurement Techniques*, S. G Malhotra, Z. U. Rek, S. M. Yalisove, J. C. Bilello, Thin Solid Films, **301** (1997) 45-54.

6. *Depth-Sensitive Strain Analysis Of A W-Ta-W Trilayer*, S. G Malhotra, Z. U. Rek, S. M. Yalisove, J. C. Bilello, Thin Solid Films **301**, (1997) 55-61.

7. *Strain Gradients And Normal Stresses In Textured Mo Thin Films*, S. G. Malhotra, Z. U. Rek, S. M. Yalisove, J. C. Bilello, J. Vac. Sci. Technol. **A15** (1997) 345-352.

8. Polycrystalline Thin Films, edited by S.M. Yalisove, B.L. Adams, J.S. Im, Y. Zhu, and F.R. Chen, (Mater. Res. Soc. Proc. **472** Pittsburgh, PA, 1997).

9. *An In-situ/Ex-situ X-ray Analysis System for Thin Sputtered Films*, A. K. Malhotra, J. F. Whitacre, Z. B. Zhao,J. Hershberger, SM Yalisove and J. C. Bilello, Surf. Coat. Tech. **110** (1998) 105-110).

10. *Surface Roughness and In-Plane Texturing in Sputtered Thin Films,* J. F. Whitacre, Z. U. Rek, J. C. Bilello and S. M. Yalisove, J. Appl. Phys. **84**(3) (1998) 1346-1353.

11. *Stresses in Multilayered Thin Films,* R. C. Cammarata, J. C. Bilello, A. L. Greer, K. Sieradzki, and S. M. Yalisove, MRS Bulletin, **24**(2), (1999) 34-38.

Ph. D. Theses:

1. *Depth Profiling of Strain in Thin films and Multilayers*, Ph. D. Thesis, University of Michigan, 1996 Sandra Guy Malhotra, **(J. C. Bilello and S. M. Yalisove** co-advisors).

2. *Evolution of Surface Roughness and Texture During Low Temperature Film Deposition*, Ph. D. Thesis, University of Michigan, 1996 Oleh Petro Karpenko, **(S. M. Yalisove** advisor).

3. *The Nature of Stress in Nanoscale Refractory Metal Sputtered Films*, Ph. D. Thesis, University of Michigan, 1997 Laraba Jean Parfitt, **(J. C. Bilello and Steve Yalisove** co-advisors).

4. *Strain and Structure of Amorphous Boron Carbide and Silicon Carbide Thin Films*, Ph. D. Thesis, University of Michigan, 1999 Jeffery Gerard Hershberger, **(J. C. Bilello and S. M. Yalisove** co-advisors).

Technical Reports Published in Annual Volumes:

1. *Probing In-Plane Texture Development in Sputter-Deposited Thin Mo Films Using Grazing Incidence X-ray Scatttering,*J. F. Whitacre, O. P. Karpenko, Z. U. Rek, S. M. Yalisove and J. C. Bilello, Activity Report for the Stanford Synchrotron Radiation Laboratory, Proposal no. 2346MP, (1996).

2. *Structure of Sputter Deposited Boron Carbide and Silicon Carbide Thin Films,*J. Hershberger, F. Kustas,, Z. U. Rek, L. Fehrenbacher, S. M. Yalisove and J. C. Bilello, Activity Report for the Stanford Synchrotron Radiation Laboratory, Proposal no. 2346MP, (1996).

3. *Phase and Stress State in Ultra Thin Ta Films Via Grazing Incidence X-Ray Scattering,* J. F. Whitacre, Z. U. Rek, S. M. Yalisove, and J. C. Bilello, (1997) SSRL Activity Report, Proposal 2346MP.

4. *Film Texturing on Rough Surfaces,* J. F. Whitacre, Z. U. Rek, J. C Bilello and S. M. Yalisove, (1997) SSRL Activity Report, Proposal 2346MP

5. *Structure of an Amorphous Thin Buried Layer*, J. Hershberger, Z. U. Rek, S. M. Yalisove, J. C. Bilello, (1997) Stanford Synchrotron Radiation Laboratory Activity Report, Proposal 2346MP.

6. *Strain Tensors in Amorphous Thin Films,* J. Hershberger, Z. U. Rek, S. M. Yalisove, J. C. Bilello, (1997) Stanford Synchrotron Radiation Laboratory Activity Report, Proposal 2346MP.

Conference and Workshop Presentations:

1. *Depth dependence of residual strains in textured Mo thin films using high resolution x-ray diffraction*, S. G. Malhotra, Z. U. Rek, S. M. Yalisove, and J. C. Bilello, in Polycrystalline Thin Films: Structure, Texture, Properties and Application II, MRS Symposium Proceedings, Vol. **403**, (1996), 127-132.

2. *Origin of in-plane texturing in sputtered Mo films,* A. K. Malhotra, S. M. Yalisove, and J. C. Bilello, in Polycrystalline Thin Films: Structure,

Texture, Properties and Application II, MRS Symposium Proceedings, Vol. **403**, (1996), 33-38.

3. *Explaining the Development of In-Plane Texture on Roughened Surfaces,* J. F. Whitacre, J. C. Bilello, O. P. Karpenko and S. M. Yalisove, MRS Symposium on: Polycrystalline Thin Films III - Structure, Texture, Properties, and Applications, , Boston, MA, December, 1996.

4. *The Role of Roughness in Texture Development*, J. F. Whitacre, J. C Bilello and S. M. Yalisove, Accepted for publication: MRS Symposium Proceedings on: Structure and Evolution of Surfaces, Vol. **441**, (1997).

5. *Origins Of Residual Stress In Mo And Ta Films: The Role Of Impurities, Microstructural Evolution, And Phase Transformations*, L. J. Parfitt, O. P. Karpenko, Z. U. Rek, S. M. Yalisove and J. C. Bilello, MRS Symposium Proceedings, Vol. **436**, (1997), 505-520.

6. *Native Oxide and the Residual Stress of Thin Mo and Ta Films,* L. J. Parfitt, Z. U. Rek, S. M. Yalisove and J. C. Bilello, MRS Symposium Proceedings, Vol. **441**, (1997).

7. *On the Measurement of Residual Stress in Thin Films*, Z. B. Zhao, J. G Hershberger, S. M. Yalisove and J. C. Bilello, Mat. Res. Soc. Symp: Stress and Mechanical Properties in Thin Films, Vol. **505**, (1998), 519-525.

8. *Testing a Model Used to Describe In-Plane Texturing In Sputtered Mo Films*. J. F. Whitacre, Z. U. Rek, J. C. Bilello, and S M. Yalisove, Materials Research Society, December 1997 Meeting, Boston MA, Symposium A: Evolution of Surface Morphology and Thin-Film Micro-structure.

9. *Real-Time In-Situ X-Ray Diffraction Studies of Sputter Deposited Thin Films*, J. F. Whitacre, S. M. Yalisove, and J. C. Bilello, Materials Research Society, December 1997 Meeting, Boston MA, Symposium A: Evolution of Surface Morphology and Thin-Film Microstructure.

10. *Structure determination of B4C and SiC thin films via synchrotron high-resolution diffraction,* J. Hershberger, F. Kustas, Z. U. Rek, S. M. Yalisove and J. C. Bilello, Mat. Res. Soc. Symp. - Thin

Films-Stresses and Mechanical Properties, Vol. **505**, (1998), 635-640.

11. *Degree of Crystallinity and Strain in B4C and SiC thin films as a function of processing conditions,* J. Hershberger, F. Kustas, Z. U. Rek, S. M. Yalisove and J. C. Bilello, Mat. Res. Soc. Proceedings, Symp. Vol. 524: Application of Synchroron Radiation Techniques to Materials Science, **Vol. 524**, (1998), 109-114.

12. *Probing Stress State and Phase Content in Ultra-Thin Ta Films,* J. F. Whitacre, Z. U. Rek, J. C. Bilello and S. M. Yalisove, Mat. Res. Soc. Proceedings, Symp.- Applications of synchrotron radiation to materials science Mat. Res. Soc. Symp. Proc. San Francisco, CA, Vol. **524**, (1998), 115-119.

Invited Presentations:

1. *Texture of Evolving Films*, S.M. Yalisove, Army Research Office Workshop on: Hot Gas Erosion and Wear of Materials, Webber's Inn, Ann Arbor, MI, September 25-27, 1996.

2. *Joy of Stress*, J. C. Bilello, Army Research Office Workshop on: Hot Gas Erosion and Wear of Materials, Webber's Inn, Ann Arbor, MI, September 25-27, 1996.

3. *Evolution of In-Plane Texture During Thin Film Deposition*, S. M. Yalisove, Workshop on Dynamics of Crystal Surfaces and Interfaces Traverse City, August 7, 1996

4. *Process Control During Growth of Thin Films and Multilayers,*. S. M. Yalisove, Seminar at: Air Force Materials Laboratory, Wright-Patterson Air Force Base, Dayton, OH, November 14, 1996.

5. *Stress Control in Thin Film Growth,*. J. C. Bilello, Seminar at: Air Force Materials Laboratory, Wright-Patterson Air Force Base, Dayton, OH, November 14, 1996.

6. *Metallization Texture,* S. M. Yalisove, Seminar Department of Materials Science and Engineering, University of Michigan, Ann Arbor, MI, January, 1997

7. *Designer Coatings for Tribological Applications*, J. C. Bilello, S. M. Yalisove, F. Kustas, and L. Fehrenbacher, Keynote talk at Tribology section of the International Conference on Metallurgical Coatings and Thin Films, San Diego, CA, April 21, 1997.

8. *Evolution of Microstructure and Strain During Sputter Deposition of Polycrystalline Thin Films*, S. M. Yalisove, Bell Laboratories, Murray Hill NJ, November 11, 1997.

9. *Mass Transport and In-Plane Texturing During Low Temperature Sputter Deposition of Metallic Films*, S. M. Yalisove, MRS fall meeting, Boston MA, December 2, 1997.

10. *Microstructure Evolution during Energetic Growth*, S. M. Yalisove, Gordon Conference, Kimball Academy, August 21, 1997

11. *Texturing on Amorphous Substrates During Sputtering*, S. M. Yalisove, Max Plank Institute, Stuttgart, Germany, July 24, 1997.

12. *Self-Orientation of Grains During Sputter Deposition*, S. M. Yalisove, Oxford University, Oxford, England, July 14, 1997.

13. *Texturing Of Polycrystalline Thin Films During Low Temperature Growth*, S. M. Yalisove, IMEC, Leuven Belgium, June 27, 1997

14. *Texture and Strain Evolution During Sputter Deposition*, S. M. Yalisove, TU Delft, Delft, Netherlands, June 18, 1997.

15. 1997*Texture, its evolution during sputter deposition*, S. M. Yalisove, Seminar at: FOM Institute for Atomic and Molecular Physics, Amsterdam, Netherlands, May 26, 1997.

16. *Evolution of thin film textures in Mo films*, S. M. Yalisove, in the symposium on Advances in Coatings Technologies II during the TMS winter meeting, February 1997.

17. *High Resolution Strain Profiling of Superconducting Thin Films*, J. C Bilello, at the Department of Engineering Sciences, University of Oxford, Parks Road, Oxford, UK, November 12, 1997

18. *Evolution of Stress and Texture in Thin Film Nanostructures*, J. C Bilello, Seminar at: North Carolina State University, Raleigh, NC, January 10, 1997 (Note: This presentation was also put on North Carolina State Public Television).

19. *Coping with Stress and Texture in Thin Film Nanostructures*, J. C Bilello, Seminar at: University of Wisconsin-Milwaukee, Milwaukee, WI, April 15, 1997.

20. *Stress in Thin Films*, J. C. Bilello, Seminar at: FOM Instituut voor Atoom- en Molecuulfysica, Amsterdam, Netherlands, May 23, 1997

21. *Controlling Texture During Sputter Deposition,* S. M. Yalisove, Department of Materials Science and Engineering, University of California at Los Angeles, Westwood, CA, May 8, 1998.

22. *Everything You Wanted to Know About Stress But were Afraid to Ask*, John C. Bilello, Department of Materials Science and Engineering, University of California at Los Angeles, Westwood, CA, February 27, 1998.

23. *High Resolution Synchrotron Radiation Studies of Structural Thin Films*, J. C. Bilello, Invited Keynote Talk at Stanford Synchrotron Radiation Laboratory Annual User Meeting, Menlo Park, CA, October 15, 1998.

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